\* ·\* O 22 12) May

Summary Report No. II Covering the Period April 22, 1970 to June 30, 1971 June 30, 1971

# **DETERMINATION OF CONTAMINATION** CHARACTER OF MATERIALS IN SPACE TECHNOLOGY TESTING

DANIEL L. HAYNES and DALE M. COULSON

Prepared for:

GODDARD SPACE FLIGHT CENTER NATIONAL AERONAUTICS AND SPACE ADMINISTRATION GLENN ROAD GREENBELT, MARYLAND 20771

Attn: MR. HAROLD SHAPIRO, CODE 332 TECHNICAL OFFICER

(NASA-CR-122374) CONTAMINATION CHARACTER OF MATERIALS IN SPACE TECHNOLOGY TESTING Summary Report, 22 Apr. 1970 - 30 Jun. 1971 (Stanford 71 p HC \$5.75 CSCL 11D

N73-19556

Unclas 17119

G3/18

JAN 1972



STANFORD RESEARCH INSTITUTE Menlo Park, California 94025 · U.S.A.

Summary Report No. II

Covering the Period April 22, 1970 to June 30, 1971

June 30, 1971

# DETERMINATION OF CONTAMINATION CHARACTER OF MATERIALS IN SPACE TECHNOLOGY TESTING

By: DANIEL L. HAYNES and DALE M. COULSON

Prepared for:

GODDARD SPACE FLIGHT CENTER
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GLENN ROAD
GREENBELT, MARYLAND 20771

Attn: MR. HAROLD SHAPIRO, CODE 332 TECHNICAL OFFICER

CONTRACT NAS5-11697

SRI Project PSU-7907

Approved by:

DALE M. COULSON, Senior Scientist

E. C. WOOD, Executive Director SRI-Irvine

Copy No. .....2....

### CONTENTS

LIST OF ILLUSTRATIONS ii
LIST OF TABLES
INTRODUCTION
EXPERIMENTAL APPARATUS AND PROCEDURES
RESULTS AND DISCUSSION
Thermofit RNF-100
Insulated Wire - TRT-24-19-V-93
Eccofoam FS
Moxness MS 60 SO8
Raychem Wire - 44/0411
Polyimide Tape X1156
Insulgrease G-640
Dow Corning High Vacuum Grease
Scotch Pressure-Sensitive Tape No. Y-9050, 3M Company 3
Eccofoam FPH, Emerson & Cuming, Inc
RTV-41 Liquid Silicone Rubber, General Electric 4
RTV-577 Liquid Silicone Rubber, General Electric 4
RTV-602 Liquid Silicone Rubber, General Electric 5
Epon 934 - Epoxy Adhesive, Shell Chemical Co 5
Stycast 1090 - Epoxy Foam, Emerson & Cuming, Inc 5
Epon 828 - Epoxy Adhesive, Shell Chemical Co 6
FUTURE WORK

### ILLUSTRATIONS

Figure	1	Thermal-Vacuum Chamber	3
Figure	2	Distribution of Outgases in Thermal-Vacuum Apparatus	5
Figure	3	Materials Subjected to Thermal Vacuum Treatment 10-1	3
Figure	4	Reflectivity of a Mirror Contaminated with Outgases from Polyolefin Shrinkable Tubing, Thermofit RNF-100	4
Figure	5	Reflectivity of a Mirror Contaminated with Outgases from Polyolefin Shrinkable Tubing RNF-100, Chill-Plate Cold	.5
Figure	6	Reflectivity of a Mirror Contaminated with Outgases from Polyolefin Shrinkable Tubing RNF-100, after Heating Chill-Plate	6
Figure	7	Microbalance Results, Thermofit RNF-100	
Figure	8	Quadrupole Mass Spectrum for Outgases from Polyolefin Shrinkable Tubing, Thermofit RNF-100 (sample heater on)	
Figure	9	Quadrupole Mass Spectrum for Outgases from Polyolefin Shrinkable Tubing, Thermofit RNF-100 (chill-plate heater on)	0
Figure	10	Pressure and Microbalance Results— Insulated Wire-TRT-24-19-V-93	1
Figure	11	Reflectivity of a Mirror Contaminated with Outgases from Insulated Wire—TRT-24-19-V-93 2	2
Figure	12	Quadrupole Mass Spectrum for Outgases from Insulated Wire—TRT-24-19-V-93 (sample heater on)	3
Figure	13	Quadrupole Mass Spectrum for Outgases from Insulated Wire—TRT-24-19-V-93 (chill-plate heater on)	1
Figure	14	Pressure and Microbalance Results—Eccofoam FS 2	
Figure			
Figure	16	Quadrupole Mass Spectrum for Outgases from Eccofoam FS (sample heater on)	8
Figure	17	Quadrupole Mass Spectrum for Outgases from Eccofoam FS (chill-plate heater on)	9

# ILLUSTRATIONS (Continued)

Figure	18	Pressure and Microblance Results— Moxness MS 60 808
Figure	19	Reflectivity of a Mirror Contaminated with Outgases from Moxness Material MS 60 S08
Figure	20	Pressure and Microbalance Results— Raychem Wire 44/0411
Figure	21	Reflectivity of a Mirror Contaminated with Outgases from Raychem Wire 44/0411
Figure	22	Pressure and Microbalance Results— Polyimide Tape X1156
Figure	23	Reflectivity of a Mirror Contaminated with Outgases from Polyimide Tape X1156
Figure	24	Pressure and Microbalance Results— Insulgrease G-640
Figure	25	Reflectivity of a Mirror Contaminated with Outgases from Insulgrease G-640
Figure	26	Pressure and Microblance Results— DC High Vacuum Silicone Grease
Figure	27	Reflectivity of a Mirror Contaminated with Outgases from Dow Corning High Vacuum Silicone Grease. 4:
Figure	28	Reflectivity of a Mirror Contaminated with Outgases from 3M Scotch Tape Y-9050, Chill-Plate Cold
Figure	29	Reflectivity of a Mirror Contaminated with Outgases from 3M Scotch Tape Y-9050, after Heating the Chill-Plate
Figure	30	Microbalance Results—Tape No. Y-9050
Figure	31	Reflectivity of a Mirror Contaminated with Outgases from Eccofoam FPH, Chill-Plate Cold 48
Figure	32	Reflectivity of a Mirror Contaminated with Outgases from Eccofoam FPH, after Heating the Chill-Plate to 80°C for One-Half Hour
Figure	33	Microbalance Results—Eccofoam FPH with Catalyst 12-4H
Figure	34	Reflectivity of a Mirror Contaminated with Outgases from G.E. RTV-41, Chill-Plate Cold 49
Figure	35	Reflectivity of a Mirror Contaminated with Outgases from G.E. RTV-41, after Chill-Plate
		Was Heated at 80°C for One-Half Hour

## ILLUSTRATIONS (Concluded)

51

Figure	37	Reflectivity of a Mirror Contaminated with Outgases from G.E. RTV-577	52
Figure	38	Microbalance Results—G.E. RTV-577	53
Figure	39	Reflectivity of a Mirror Contaminated with Outgases from G.E. RTV-602	55
Figure	40	Microbalance Results—G.E. RTV-602	56
Figure	41	Reflectivity of a Mirror Contaminated with Outgases from Shell Epon 934	57
Figure	42	Microbalance Results—Shell Epon 934	58
Figure	43	Reflectivity of a Mirror Contaminated with Outgases from Emerson & Cuming, Inc., Stycast 1090	59
Figure	44	Microbalance Results—Emerson & Cuming, Inc., Stycast 1090	30
Figure	45	Reflectivity of a Mirror Contaminated with Outgases from Shell Epon 828	32
Figure	46	Microbalance Results—Shell Epon 828	33

### TABLES

Table	Ι	Materials	Subjected	to	Thermal	Vacuum	Treatment	•	•	•	•	4
Table 1	Т	Materials	for Future	- S	tudy .							65

### INTRODUCTION

This report summarizes the second year's work and experience in a study of the contamination characters of selected materials used in space technology testing. Specific materials were subjected to a thermal vacuum environment, and the outgases were collected on a cold test mirror surface. Approximately one-half of the surface of the mirror was subjected to uv irradiation while the outgases were being deposited on the mirror. The purpose of these experiments was to determine the effect of uv irradiation on the contaminative character of outgases from selected materials.

The degree of contamination was measured in terms of degradation of the optical properties of the test mirror and the amount of deposit per unit area on quartz crystal microbalances placed near the test mirror, and by means of quadrupole mass spectral measurements of outgases from the test samples.

### EXPERIMENTAL APPARATUS AND PROCEDURES

The thermal-vacuum apparatus used in these tests was constructed during the study. Although the basic apparatus was described in Bi-monthly Progress Report No. 8, it is described below for convenience. Figure 1 is a drawing of the vacuum chamber mounted on the McPherson Model 225 spectrophotometer. The chill-plate on which the test mirror and the quartz crystal microbalances are mounted can be shifted from side-to-side by means of a gear drive through the top of the chamber in order to observe the uv irradiated or nonirradiated portions of the mirror. The uv source is so directed that only half of the mirror is irradiated during the thermal vacuum tests. The sensor of one microbalance is irradiated, and that of the other is not.

In the earlier experiments, which included insulated wire (TRT-24-19-V-93, Eccofoam FS) and in preliminary experiments on polyolefin shrinkable tubing (Thermofit RNF-100), a sample holder was used that was well focused on the center of the test mirror. The flux of outgases was therefore many times higher on the mirror than it was on the quartz crystals of the microbalances. This led to deposition of a much thicker film of contaminants on the mirror than on the balance crystals, making it difficult to correlate the microbalance results with the weight of the deposit on the mirror.

The remainder of the experiments on the materials listed in Table I were made with a sample holder that distributes the outgases through a wider angle. Figure 2 gives an approximate curve for the flux of outgases at the mirror surface and at the microbalance surface using the second sample holder. If all the outgases reached the mirror, the flux would be approximately 1.0. This represents the outgases from the sample passing through a 4-sq-in area. It can be seen from this curve that the flux at the mirror is at least 5 times as great as it is at the microbalance crystals. Thus, the microbalance results should probably be multiplied by approximately 5 to get the average deposit intensities that would be found on the mirror.

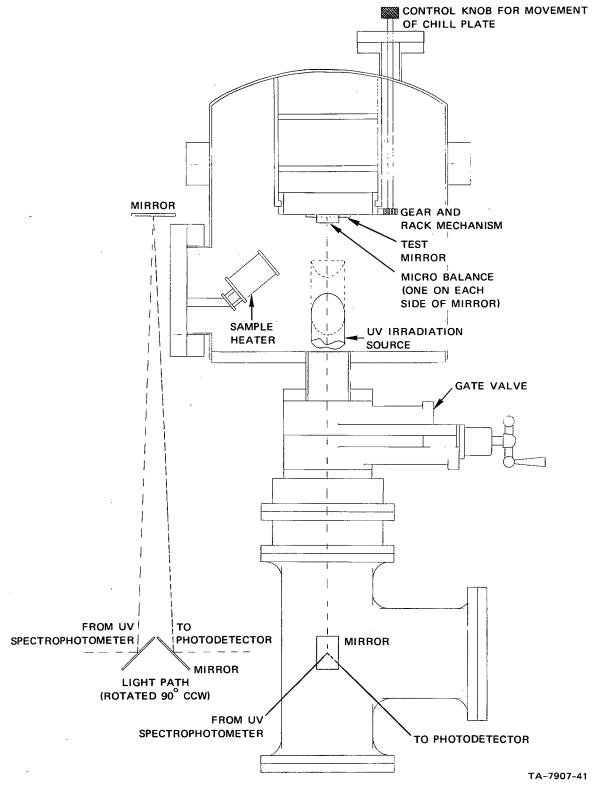


FIGURE 1 THERMAL-VACUUM CHAMBER

Table I

# MATERIALS SUBJECTED TO THERMAL VACUUM TREATMENT

Material Identification	Manufacturer	Description
Thermofit RNF-100	Raychem Corp.	Polyolefin shrinkable tubing
TRT-24-19-V-93	Raychem Corp.	Insulated wire
Eccofoam FS	Emerson & Cuming, Inc.	Polyurethane foam
Moxness MS60 508	Moxness Products, Inc.	Silicone elastomer
Raychem wire 44/0411	Raychem Corp.	Insulated wire
Polyimide tape X1156 (electrical tape No. 92)	3M Company	Polyimide film with thermosetting silicone pressure-sensitive tape
Insulgrease G-640	General Electric	Dielectric silicone grease
High vacuum silicone grease	Dow Corning	Silicone lubricant
Scotch tape No. Y-9050	3M Company	Pressure-sensitive tape
Eccofoam FPH	Emerson & Cuming, Inc.	Polyurethane foam
RTV-41	General Electric	Liquid silicone rubber
RTV-577	General Electric	Liquid silicone rubber
RTV-602	General Electric	Liquid silicone rubber
Epon 934	Shell Chemical Co.	Epoxy adhesive
Stycast 1090	Emerson & Cuming, Inc.	Epoxy foam
Epon 828	Shell Chemical Co.	Epoxy adhesive

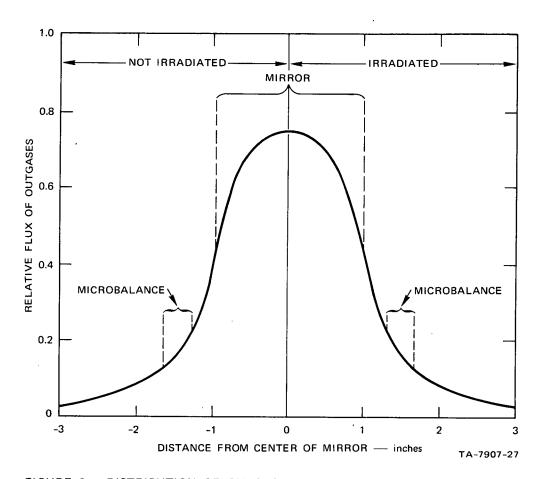


FIGURE 2 DISTRIBUTION OF OUTGASES IN THERMAL-VACUUM APPARATUS

A second factor that makes it difficult to correlate the microbalance results with thickness of deposits on the mirror is that the mirror was held at a colder temperature than the crystals of the microbalance because of poor thermal contact between the crystals and the chill-plate. Thus, condensation was probably more efficient on the mirror than on the crystals. This is particularly true of the irradiated crystal because of irradiative heating.

Not shown in Figure 1 are the ionizer section of a quadrupole mass spectrometer, which is placed in a port in the wall of the main chamber but not in line-of-sight of the test sample, the sensor of a CVC Type GIC-110 ionization vacuum gage, and other auxiliary ports. The chill-plate is cooled by means of refrigerant from an external refrigerator charged with monochlorodifluoromethane.

The uv source gives the following intensities at the surface of the mirror:

$\underline{\text{Wavelength }}(\mathring{A})$	Intensity (watts/cm <sup>2</sup> )
2753	0.02
2804/5/6	0.04
2894	0.11
2968	0.23
3023/6	0.33
3650	0.81

The experimental procedure was as follows. A 1-g sample of finely divided material was placed in the sample holder. The vacuum system was sealed, and it was evacuated with a diffusion pump charged with Dow Corning 705 diffusion pump fluid. The pressure dropped rapidly to the  $10^{-6}$  torr range. The system was pumped overnight or at least for several hours until the chamber pressure was approximately  $2 \times 10^{-7}$  torr.

The gate valve to the McPherson spectrophotometer was opened, and the chamber pressure increased to the  $10^{-5}$  torr range. The spectral reflectivity for the wavelength range of 1000 to 6000 Å was measured on two areas of the mirror. The two parts of the mirror scanned represent

mercury lamp irradiated and nonirradiated areas during the experiment. The gate valve was closed, and the chamber pressure dropped to the  $10^{-7}$  torr range.

The chill-plate refrigerator was turned on, and the chill-plate that holds the test mirror cooled to -10 to  $-20^{\circ}\text{C}$  in approximately 20 minutes.

Mass spectra of the chamber gases were determined with a quadrupole mass spectrometer before, and at numerous times during the experiment to gather information concerning the molecular species emitted from the sample.

Chamber pressure readings were made with a Consolidated Vacuum Corporation Type GIC-110 ionization vacuum gage as often as necessary to define the time-pressure relationship during the experiment. Frequent readings were also taken with the quartz crystal microbalance. The exposed crystal of one of the microbalances was irradiated with the mercury lamp; the exposed crystal of the other microbalance was not irradiated.

The sample holder was heated from ambient temperature to  $125^{\circ}\text{C}$  in approximately 20 minutes. The power to the heater was then reduced to maintain the sample temperature at  $125^{\circ}\text{C}$ .

At the conclusion of the experiment the power to the sample heater was turned off and the sample holder allowed to cool. The uv irradiation source was turned off and the spectral reflectivity from 1000 to 6000  $\mathring{\text{A}}$  was measured on irradiated and nonirradiated areas of the test mirror.

Next, the chill-plate refrigerator was turned off and the chill-plate heater turned on. The chill-plate was heated to  $80^{\circ}$ C in approximately 20 minutes. During this period, mass spectral patterns on the chamber gases were measured frequently. The power to the heater was then set to hold the temperature at  $80^{\circ}$ C for 30 minutes.

Again, the reflectivity was measured on the two areas of the test mirror. The chamber was held under vacuum for a period of several hours

to allow the chill-plate to come to ambient temperature. The chamber was opened and the test mirror was removed for examination. Photographs and photomicrographs were taken to document the general appearance and nature of the deposits. Other tests were also made on the deposits to characterize them further. These tests included visible and infrared reflectivity in the 0.2- to 15-micron range, solubility of the deposit in organic solvents, elemental composition, weight, and the infrared absorption spectrum of a concentrated solution of the deposits.

### RESULTS AND DISCUSSION

Materials subjected to thermal-vacuum treatment with uv irradiation of the test mirror on which outgases deposited are listed in Table I.

Photographs of the test mirrors after removing them from the vacuum chamber, given in Figure 3, gave a good permanent record of the visual appearance of the deposits. There is a qualitative agreement between the appearance of these photographs and the other data collected in these studies.

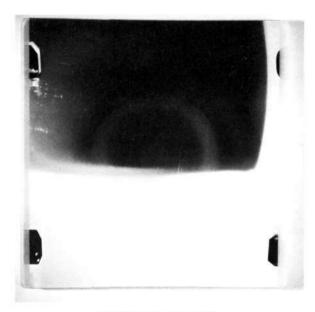
### Thermofit RNF-100

During the course of the study several tests were run on Thermofit RNF-100. Figure 4 gives reflectivity curves for uv-irradiated and non-irradiated portions of a mirror subjected to outgases of Thermofit RNF-100 for a period of 5 hours with the highly focused sample holder. The data on which these curves were based were measured with a Cary Model 14M spectrophotometer after the mirror was removed from the vacuum chamber.

Figure 5 shows reflectivity curves for RNF-100 measured with the McPherson vacuum uv spectrophotometer with the mirror still in the vacuum system and with the chill-plate cold. Since the wide-angle sample holder was used in this experiment, the outgas flux was approximately that shown in Figure 2.

Figure 6 gives reflectivity curves for the same mirror after the chill-plate was held at  $80^{\circ}\text{C}$  for 30 minutes, still in the vacuum chamber. It is apparent that this heat treatment of the contaminated mirror under vacuum did not improve the reflectivity significantly. These measurements were made after 96 hours of exposure of the mirror to outgases of a 1-g sample of RNF-100. The sample chamber was held at  $125^{\circ}\text{C}$ , and the chill-plate was held at  $-20^{\circ}\text{C}$  for the duration of the 96-hour exposure.

Figure 7 gives the microbalance results for the 96-hour experiment. It shows that the nonirradiated crystal gained more weight than the irradiated one and that deposition continued at an appreciable rate during

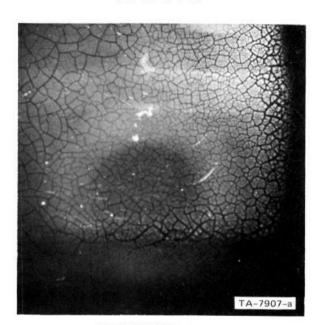


THERMOFIT RNF-100

TRT-24-19-V-93



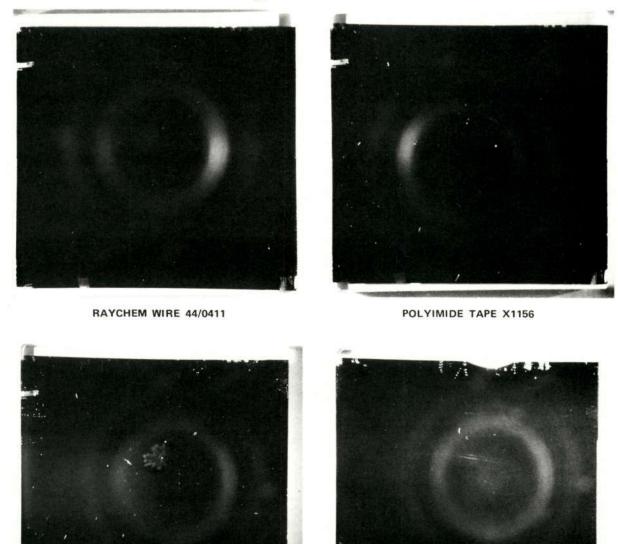




MOXNESS MS60 S08

FIGURE 3 MATERIALS SUBJECTED TO THERMAL VACUUM





**INSULGREASE G-640** 

HIGH VACUUM SILICONE GREASE

TA-7907-b

FIGURE 3 MATERIALS SUBJECTED TO THERMAL VACUUM (Continued)

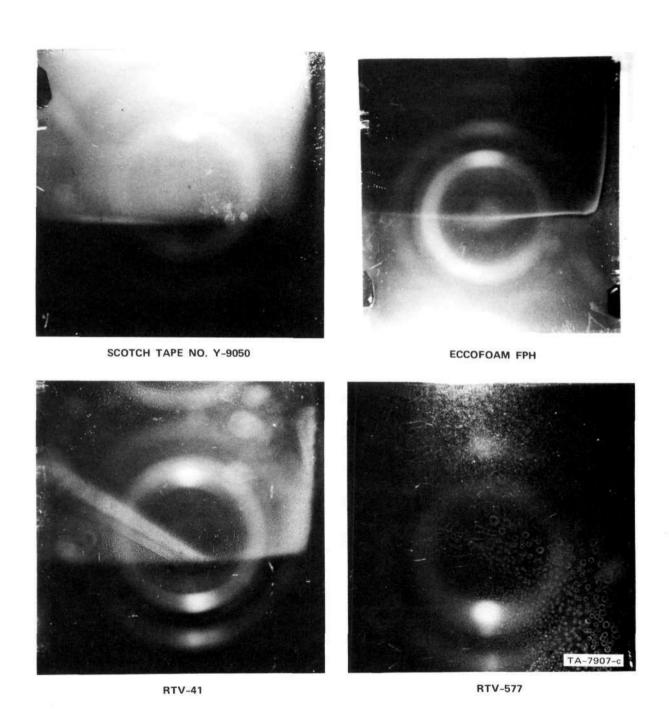


FIGURE 3 MATERIALS SUBJECTED TO THERMAL VACUUM (Continued)

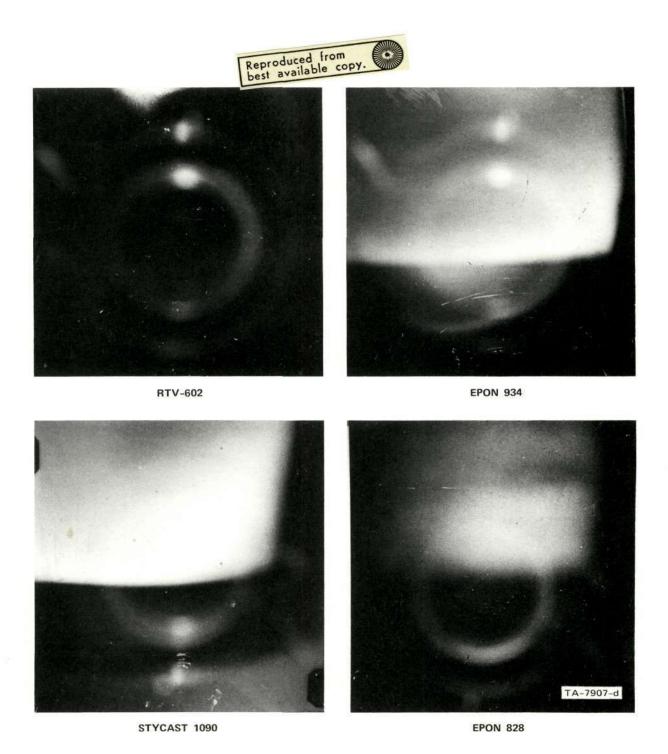


FIGURE 3 MATERIALS SUBJECTED TO THERMAL VACUUM (Concluded)

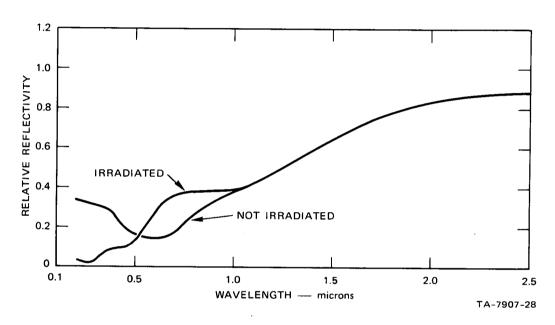


FIGURE 4 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM POLYOLEFIN SHRINKABLE TUBING, THERMOFIT RNF-100

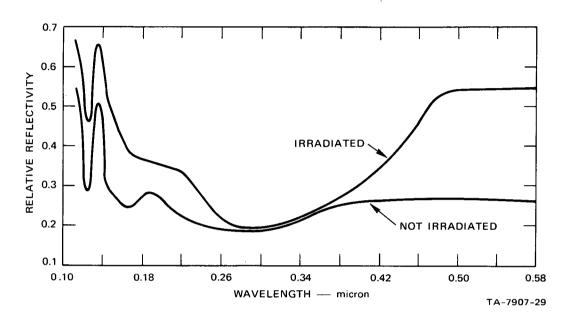


FIGURE 5 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM POLYOLEFIN SHRINKABLE TUBING RNF-100, CHILL-PLATE COLD

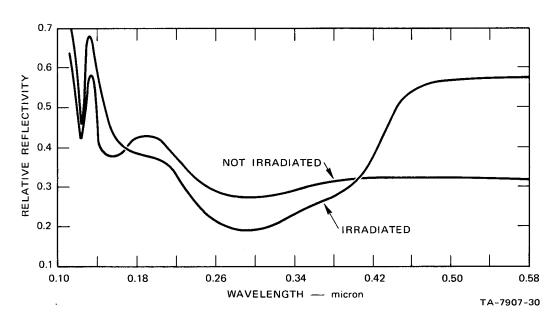


FIGURE 6 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM POLYOLEFIN SHRINKABLE TUBING RNF-100, AFTER HEATING CHILL-PLATE

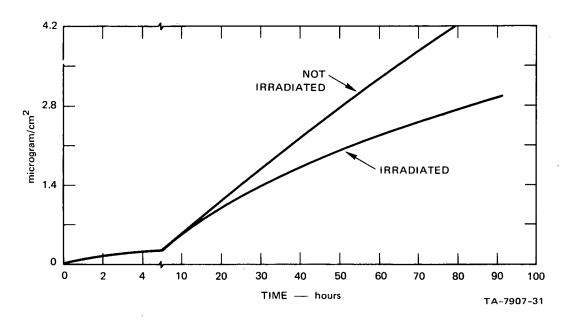


FIGURE 7 MICROBALANCE RESULTS—THERMOFIT RNF-100

the total period of the experiment. Thus, RNF-100 continues to outgas over a long period of time. The heavier deposit on the nonirradiated crystal correlates well with the visual appearance of the mirror. Visual inspection of the mirror after the experiment showed that the nonirradiated side had a heavier deposit than the irradiated side. It is estimated from the weight increases of the mirror, from the quartz crystal microbalance results, and from Figure 2 that the deposit was about 0.2 micron thick on the mirror at the end of the experiment. This material gave by far the heaviest deposit of any of the materials studied so far.

Figure 8 gives the quadrupole mass spectrum for outgases from the sample just as the sample initially reached  $125^{\circ}\text{C}$ . Figure 9 gives a quadrupole mass spectrum recorded during the time near the end of the experiment when the chill-plate had been heated from  $-30^{\circ}\text{C}$  to  $+80^{\circ}\text{C}$ . The spectra in Figures 8 and 9 are similar and probably represent predominately  $C_6$ -hydrocarbons.

### Insulated Wire - TRT-24-19-V-93

Figure 10 gives chamber pressure and microbalance readings for insulated wire, TRT-24-19-V-93. Since this experiment was run with the focused sample holder, the deposit on the mirror was unrealistically heavy. There was very little deposit on the irradiated portion of the mirror and a heavy white deposit on the nonirradiated areas. The reflectivity curves taken after the chill-plate had been held at 80°C for 30 minutes, given in Figure 11, shows this effect. Reflectivity data for wavelengths larger than 6000 Å were taken on the mirror after it had been removed from the vacuum chamber. The deposits caused no degradation of reflectivity in the 2- to 15-micron region.

Figure 12 shows the quadrupole mass spectrum of sample outgases at the time the sample heater reached  $125^{\circ}\text{C}$ . Figure 13 is the quadrupole mass spectrum for the gases emitted from the chill-plate upon reaching a temperature of  $80^{\circ}\text{C}$ .

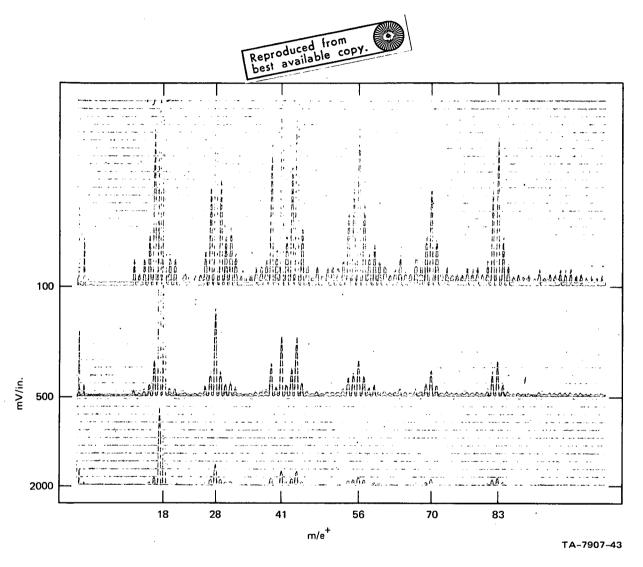
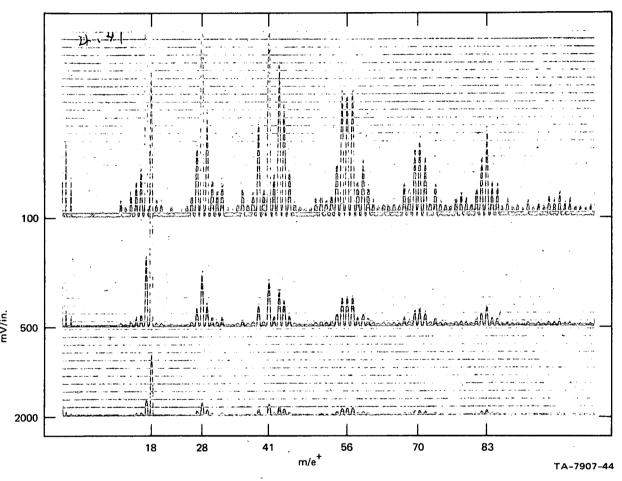


FIGURE 8 QUADRUPOLE MASS SPECTRUM FOR OUTGASES FROM POLYOLEFIN SHRINKABLE TUBING, THERMOFIT RNF-100 (sample heater on)





.FIGURE 9 QUADRUPOLE MASS SPECTRUM FOR OUTGASES FROM POLYOLEFIN SHRINKABLE TUBING, THERMOFIT RNF-100 (chill-plate heater on)

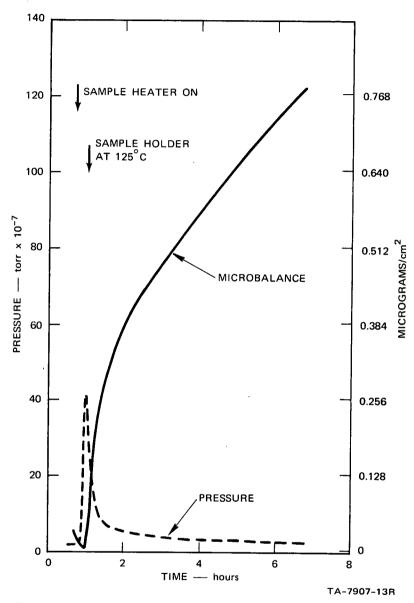


FIGURE 10 PRESSURE AND MICROBALANCE RESULTS—INSULATED WIRE-TRT 24-19-V-93

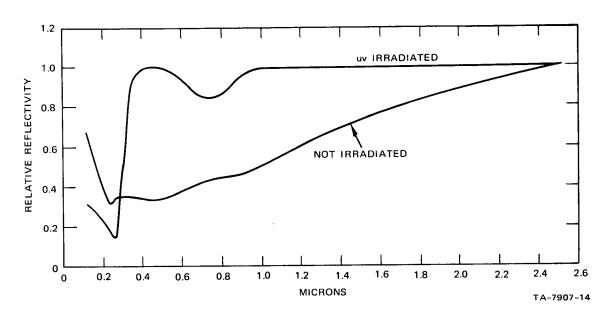


FIGURE 11 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM INSULATED WIRE—TRT 24-19-V-93

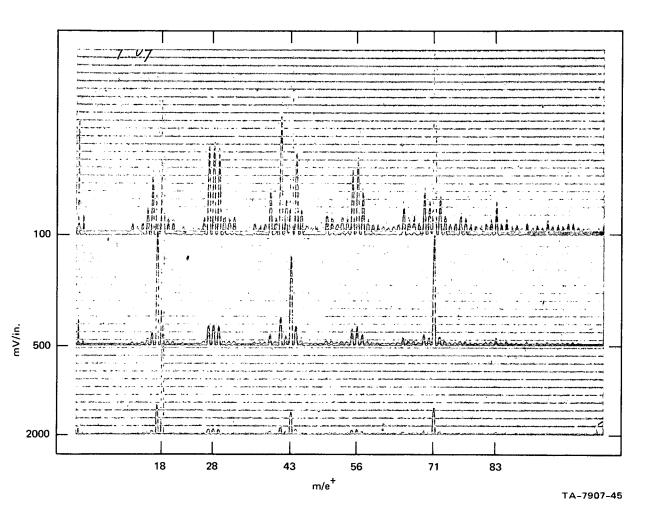


FIGURE 12 QUADRUPOLE MASS SPECTRUM FOR OUTGASES FROM INSULATED WIRE—TRT 24-19-V-93 (chill-plate heater on)

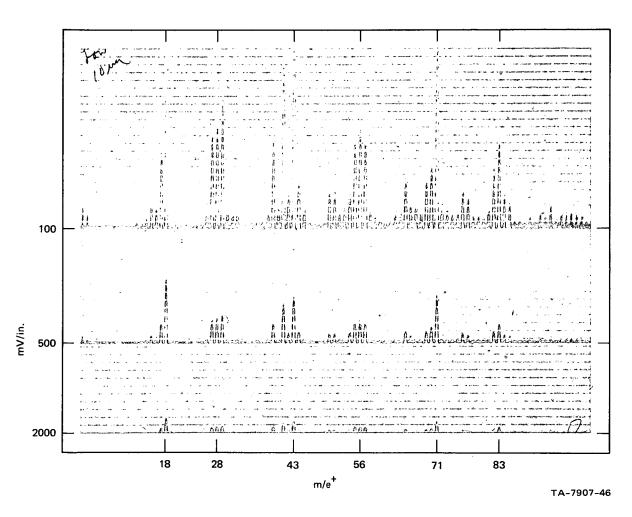


FIGURE 13 QUADRUPOLE MASS SPECTRUM FOR OUTGASES FROM INSULATED WIRE—TRT 24-19-V-93 (sample heater on)

### Eccofoam FS

Figure 14 gives pressure and microbalance results for Eccofoam FS. This was the last experiment in which the focused sample holder was used. There was a slight but visible deposit on the nonirradiated portion of the test mirror. The reflectivity curves in Figure 15 show no degradation above  $7000~\text{\AA}$ .

Figure 16 is the quadrupole mass spectrum for sample outgases when the sample heater reached  $125^{\circ}\text{C}$ . Figure 17 is the quadrupole mass spectrum of gases emitted from the chill-plate when it reached  $80^{\circ}\text{C}$ .

### Moxness MS 60 S08

Moxness material MS60 S08 was the first material tested with the nonfocused sample holder. This material gave the sample chamber pressure and microbalance curves given in Figure 18. The uv-irradiated area of the test mirror had a white, cracked appearing deposit, although the nonirradiated area appeared to be almost clean. Thickness of the deposit was in the range of 0.03 to 0.05 micron, the irradiated side of the mirror having the heaviest deposit. The reflectivity curves in Figure 19 showed relative reflectivities that were considerably above 1.0 at some wavelengths. In this case, a mirror with a very thin aluminum coating was used. This mirror exhibited considerable transmission of light in the visible region. The aluminum film was undoubtedly too thin to give good reflectivity in the uv region. Thus, the results at wavelengths shorter than 5000 Å do not give much useful information about the contaminant deposited on the mirror.

The quadrupole mass spectra for sample outgases and gases emitted from the heated chill-plate consisted of only low-molecular-weight peaks with water predominating.

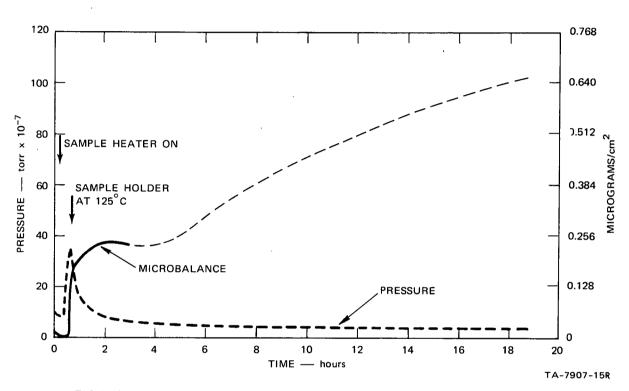


FIGURE 14 PRESSURE AND MICROBALANCE RESULTS—ECCOFOAM FS

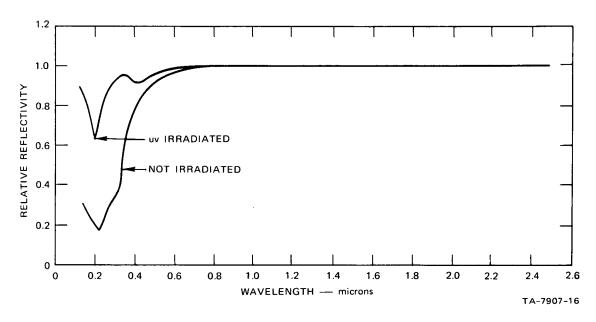


FIGURE 15 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM ECCOFOAM FS

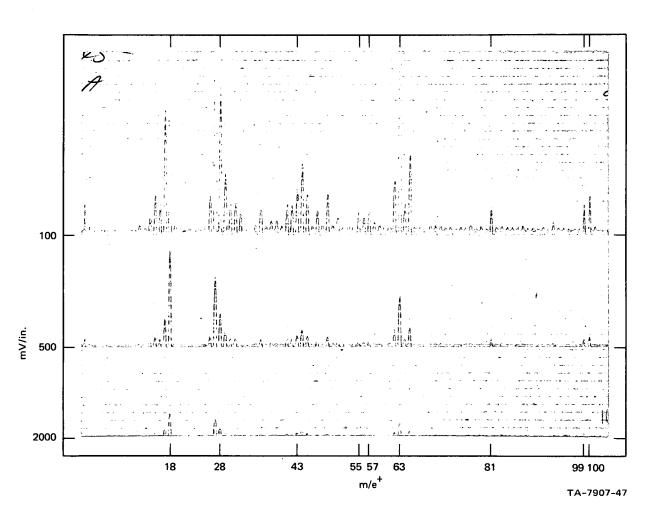


FIGURE 16 QUADRUPOLE MASS SPECTRUM FOR OUTGASES FROM ECCOFOAM FS (sample heater on)

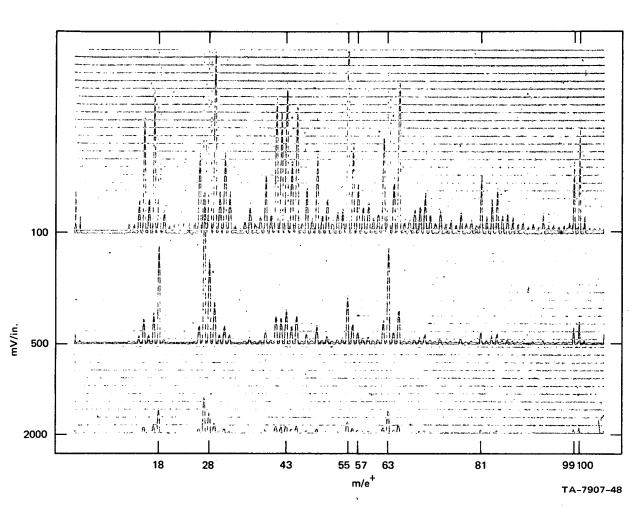


FIGURE 17 QUADRUPOLE MASS SPECTRUM FOR OUTGASES FROM ECCOFOAM FS (chill-plate heater on)

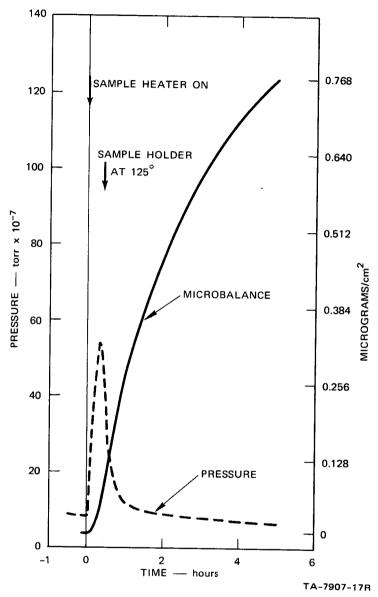


FIGURE 18 PRESSURE AND MICROBALANCE RESULTS—MOXNESS 60 S08

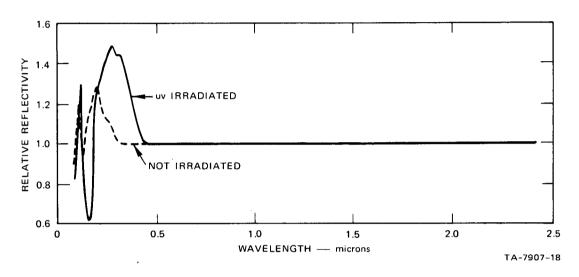


FIGURE 19 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM MOXNESS MATERIAL MS 60 S08

### Raychem Wire - 44/0411

Chamber pressure and microbalance curves for Raychem wire 44/0411 are given in Figure 20, and reflectivity curves are given in Figure 21. The kind of structure shown in Figure 21 is very similar to the theoretically derived reflectivity curves for a good aluminum reflector coated with a thin film of dielectric material. The mirror deposit was from 0.01 to 0.03 micron thick.

Except for water, no significant peaks were noted in the quadrupole mass spectra for sample outgases or gases emitted from the heated chill-plate.

# Polyimide Tape X1156

Figure 22 shows chamber pressure and microbalance curves for Polyimide Tape X1156. The hump in the microbalance curve at 1 hour after the experiment was started was probably caused by material of intermediate volatility that deposited temporarily on the quartz crystal but was gradually pumped off. Simultaneously, less volatile material was being deposited and continued to build up on the balance sensor throughout the term of the experiment. So little material was deposited on the mirror that it appeared to be clean at the end of the experiment. The mirror deposit was less than 0.01 micron thick. Figure 23 shows that there was degradation of the reflectivity only on the uvirradiated area and only at wavelengths less than 2500 Å. The non-irradiated portion of the mirror showed no degradation.

The quadrupole mass spectra of the sample outgases and gases emitted from the heated chill-plate showed water as the only significant constituent.

### Insulgrease G-640

Insulgrease G-640 gave the results shown in Figures 24 and 25. There was a slight deposit on the irradiated area. The deposit was about 0.01 to 0.03 micron thick.

Water was the main component indicated in the quadrupole mass spectra of the sample outgases and gases emitted from the heated chill-plate.

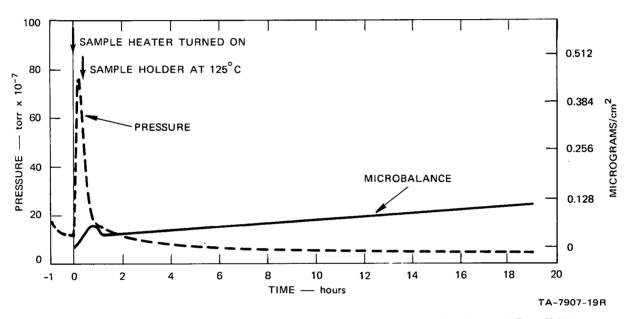


FIGURE 20 PRESSURE AND MICROBALANCE RESULTS—RAYCHEM WIRE 44/0411

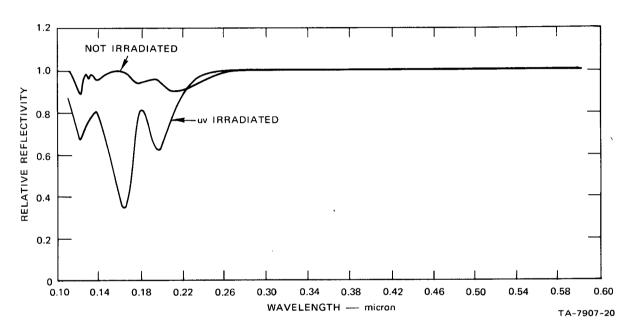


FIGURE 21 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM RAYCHEM WIRE 44/0411

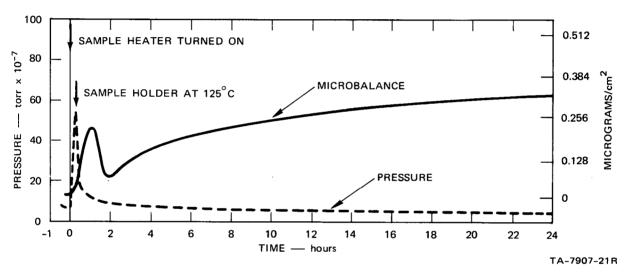


FIGURE 22 PRESSURE AND MICROBALANCE RESULTS—POLYIMIDE TAPE X1156

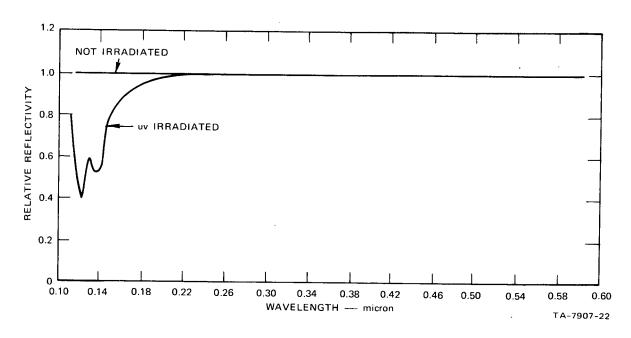


FIGURE 23 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM POLYIMIDE TAPE X1156

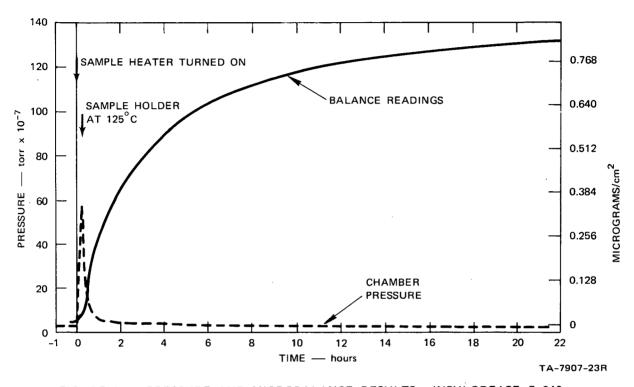


FIGURE 24 PRESSURE AND MICROBALANCE RESULTS—INSULGREASE G-640

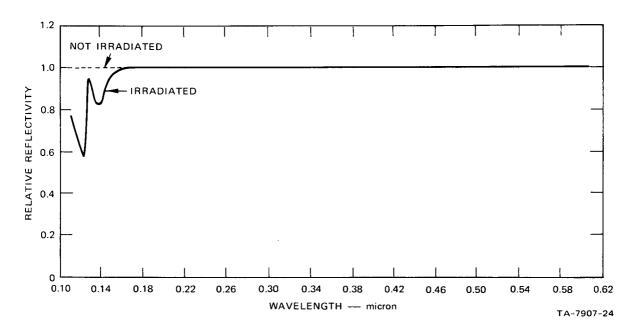


FIGURE 25 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM INSULGREASE G-640

### Dow Corning High Vacuum Grease

Dow Corning High Vacuum Silicone Grease gave the results shown in Figures 26 and 27. There was very little deposit on the mirror. It was less than 0.01-micron thick. As indicated by the lightness of the deposit on the mirror, no significant components other than water were noted in the quadrupole mass spectra of the sample outgases or gases emitted from the heated chill-plate.

## Scotch Pressure-Sensitive Tape No. Y-9050, 3M Company

A 1-g sample of Tape No. Y-9050 was subjected to a 96-hour uvirradiated thermal-vacuum experiment. Figure 28 gives reflectivity curves for irradiated and nonirradiated areas of the test mirror at the end of the test with the chill-plate still at -20°C. Figure 29 gives similar curves after the chill-plate was held at 80°C for 30 minutes. We have no explanation for the reflectivity values greater than 1.0 in the 0.16 to 0.22 micron region.

Figure 30 gives quartz crystal microbalance results for Tape No. Y-9050. In this case the deposit was heavier on the irradiated crystal and also on the irradiated side of the test mirror. When the mirror was removed from the test chamber, it appeared to be almost clean with only a slight fog on the irradiated side. The most intense deposits on the mirror in this experiment were probably no more than 0.05-micron thick.

The quadrupole mass spectral patterns were weak, showing only low-molecular-weight peaks, with water as the predominant contributor.

### Eccofoam FPH, Emerson & Cuming, Inc.

Eccofoam FPH with catalyst 12-4H was cured at 105°C for 1.5 hours and cut into slices that were approximately 1/2 in. × 1/2 in. × 3/16 in. A 1-g sample was subjected to a 96-hour, uv-irradiated, thermal-vacuum test. Reflectivity results are given in Figures 31 and 32. The deposit was definitely heavier on the nonirradiated area of the test mirror. The microbalance results in Figure 33 also show heavier deposits on the nonirradiated side. It is estimated that the deposits on the mirror were

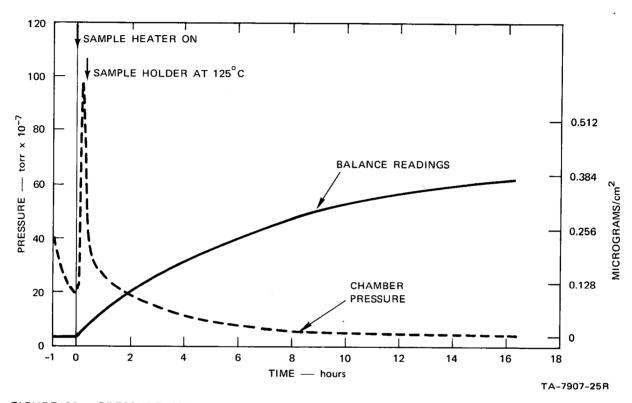


FIGURE 26 PRESSURE AND MICROBALANCE RESULTS—DC HIGH VACUUM SILICONE GREASE

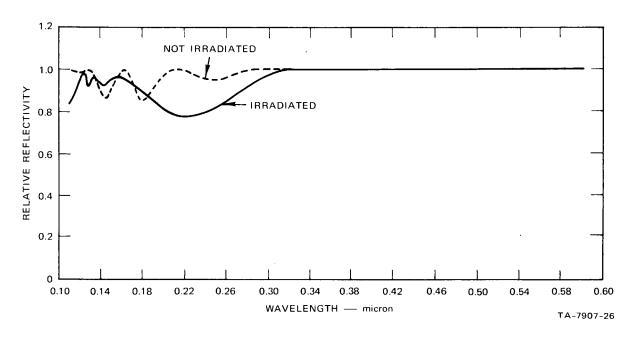


FIGURE 27 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM DOW CORNING HIGH VACUUM SILICONE GREASE

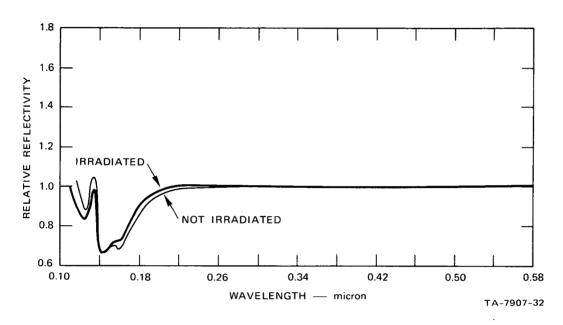


FIGURE 28 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM 3M SCOTCH TAPE Y-9050, CHILL-PLATE COLD

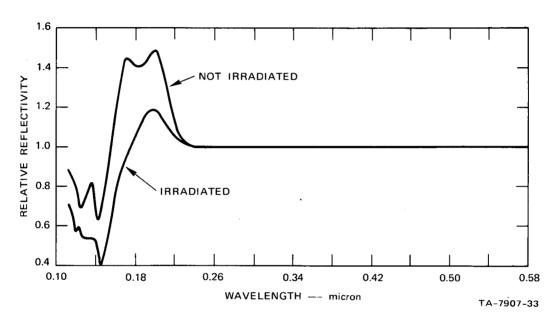


FIGURE 29 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM 3M SCOTCH TAPE Y-9050, AFTER HEATING THE CHILL-PLATE

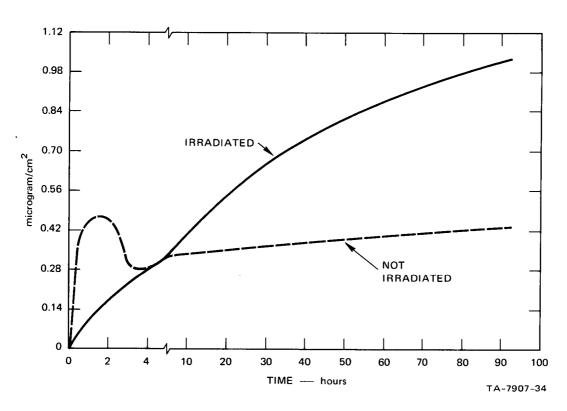


FIGURE 30 MICROBALANCE RESULTS-TAPE NO. Y-9050

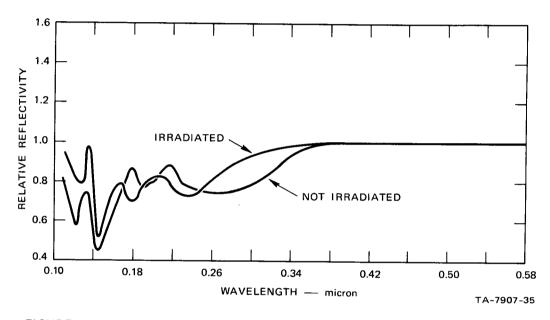


FIGURE 31 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM ECCOFOAM FPH, CHILL-PLATE COLD

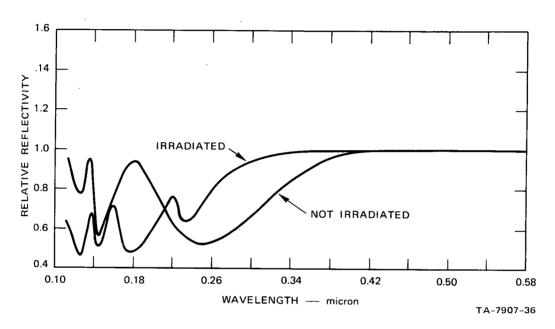


FIGURE 32 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM ECCOFOAM FPH, AFTER HEATING THE CHILL-PLATE TO 80°C FOR ONE-HALF HOUR

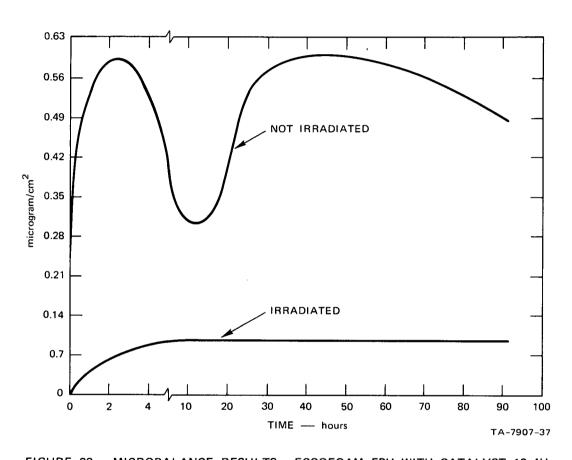


FIGURE 33 MICROBALANCE RESULTS—ECCOFOAM FPH WITH CATALYST 12-4H

no more than approximately 0.03-micron thick on the nonirradiated side and less than 0.01-micron thick on the irradiated area.

The quadrupole mass spectral patterns were very weak, showing only very low-molecular-weight peaks.

# RTV-41 Liquid Silicone Rubber, General Electric

General Electric Company's RTV-41 liquid silicone rubber was cured by the method described in Technical Data Book S-35. A 1-g sample was subjected to a uv-irradiated thermal-vacuum experiment with a 138-hour duration. Reflectivity results for the test mirror at the end of the experiment are given in Figures 34 and 35. The deposit was about 0.01 to 0.03 micron thick and was heavier on the irradiated area of the mirror. Only one microbalance functioned properly during this experiment. Figure 36 gives microbalance results for the irradiated side. A maximum was reached at about 20 hours, after which the irradiated crystal lost weight. This weight loss was probably caused by revolatilization of deposited material.

The quadrupole mass spectral curves showed no significant outgasing peaks for materials other than water.

### RTV-577 Liquid Silicone Rubber, General Electric

RTV-577 was prepared following the instructions in Technical Data Book S-35 using the 0.5% Dibutyl Tin Dilaurate procedure. A 1-g sample was subjected to a 94-hour irradiated thermal-vacuum test.

The reflectivity of the irradiated and nonirradiated sides of the mirror after the chill-plate had been heated is shown in Figure 37. Microbalance results are presented in Figure 38. The thickness of the deposit is about 0.10 to 0.15 micron and is the heaviest on the irradiated side of the mirror.

The quadrupole mass spectra of the sample outgases and gases emitted from the chill-plate at  $80^{\circ}\text{C}$  indicate water as the only significant component.

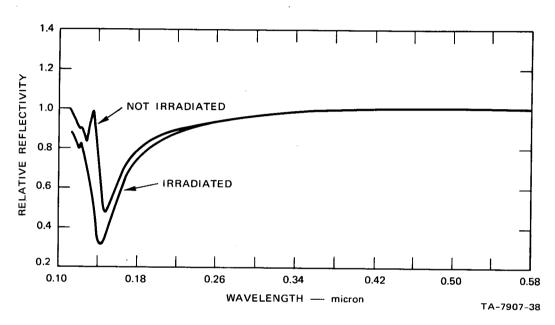


FIGURE 34 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM G.E. RTV-41, CHILL-PLATE COLD

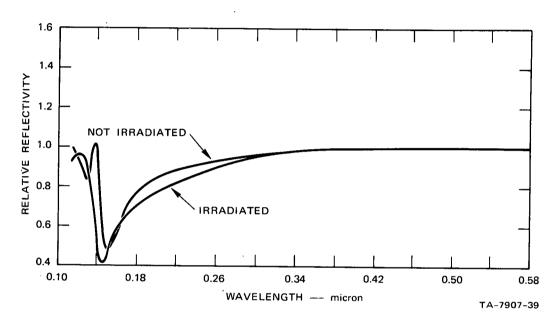


FIGURE 35 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM G.E. RTV-41, AFTER CHILL-PLATE WAS HEATED AT 80°C FOR ONE-HALF HOUR

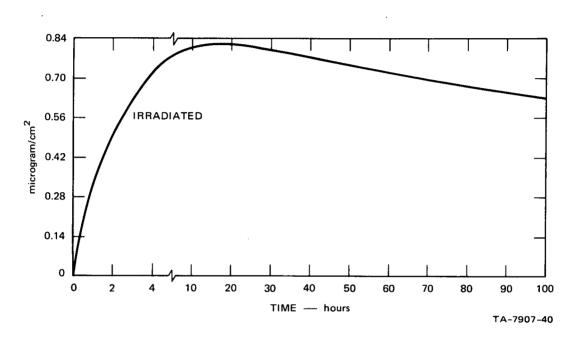


FIGURE 36 MICROBALANCE RESULTS—RTV-41

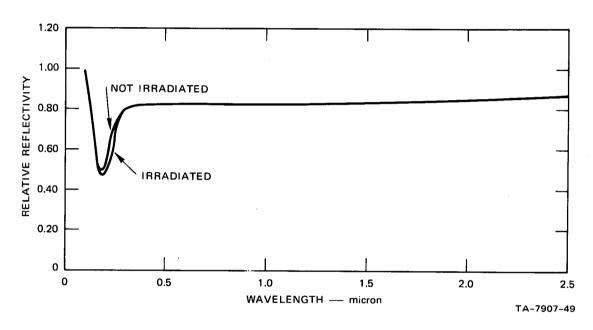


FIGURE 37 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM G.E. RTV-577

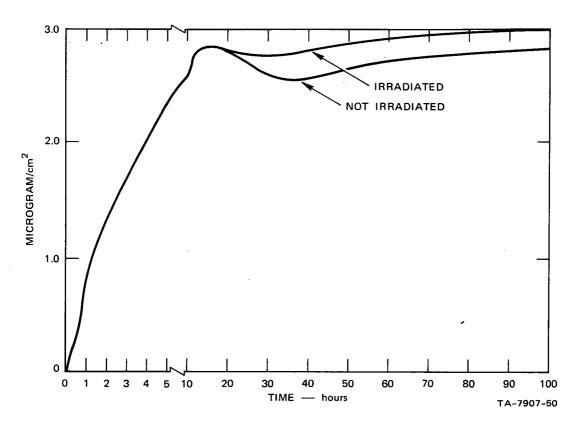


FIGURE 38 MICROBALANCE RESULTS-G.E. RTV-577

### RTV-602 Liquid Silicone Rubber, General Electric

RTV-602 was prepared using the instructions in Technical Data Book S-35 for the 0.50% SRC-05 procedure. A 70-hour irradiated thermal-vacuum test was conducted with a 1-g sample.

Figure 39 shows the reflectivity of the irradiated and nonirradiated sides of the mirror. Microbalance results are shown in Figure 40. The deposit is very light, the nonirradiated side being slightly heavier. The thickness of the deposit is no more than 0.01 micron. The heavy deposit indicated in Figure 40 was almost completely driven off by heating the chill-plate.

The quadrupole mass spectra were weak, and only water is discernible in the sample outgases and gases emitted from the heated chill-plate.

# Epon 934 - Epoxy Adhesive, Shell Chemical Co.

Epon 934 was prepared using the label instructions. A 1-g sample was used in the irradiated thermal-vacuum test. The test was run for a duration of 137 hours.

The reflectivity of the mirror is presented in Figure 41. Microbalance results are shown in Figure 42. From both graphs and from Figure 3 it can be seen that the irradiated side of the mirror has the heaviest deposit. It is estimated that the deposit is from 0.01 to 0.03 micron thick.

The quadrupole mass spectra were weak and showed no significant peaks other than water.

### Stycast 1090 - Epoxy Foam, Emerson & Cuming, Inc.

Stycast 1090 was prepared following the label instructions for the procedure using catalyst No. 9. The irradiated thermal-vacuum test was conducted with a 1-g sample for a period of 168 hours.

Reflectivity curves for the mirror are shown in Figure 43. Microbalance results are displayed in Figure 44. The mirror deposit is from 0.05 to 0.08 micron thick. Figure 43 and Figure 44 show the deposit to

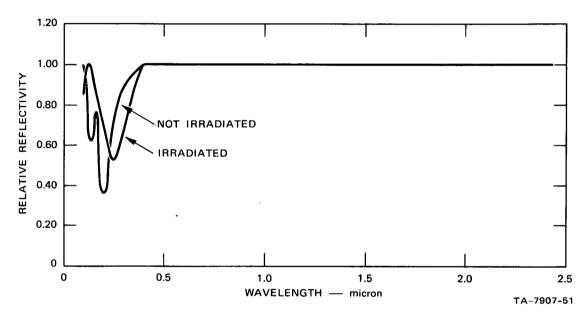


FIGURE 39 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM G.E. RTV-602

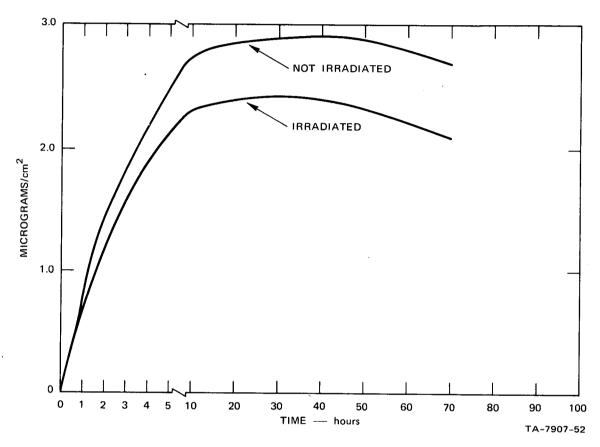


FIGURE 40 MICROBALANCE RESULTS-G.E. RTV-602

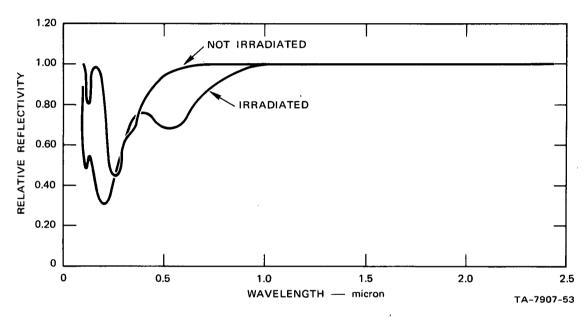


FIGURE 41 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM SHELL EPON 934

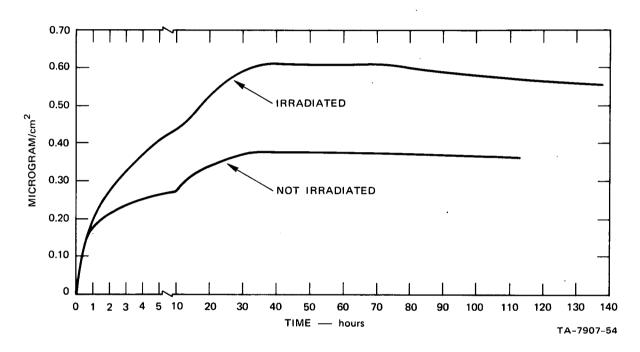


FIGURE 42 MICROBALANCE RESULTS—SHELL EPON 934

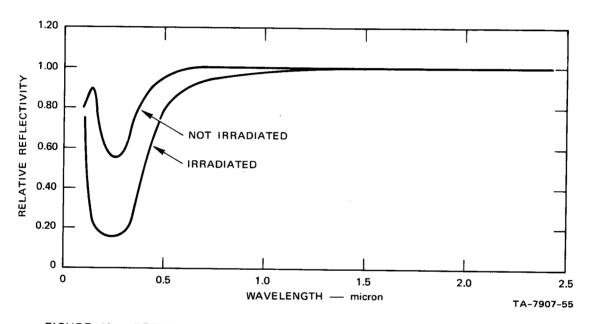


FIGURE 43 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM EMERSON AND CUMING, INC. STYCAST 1090

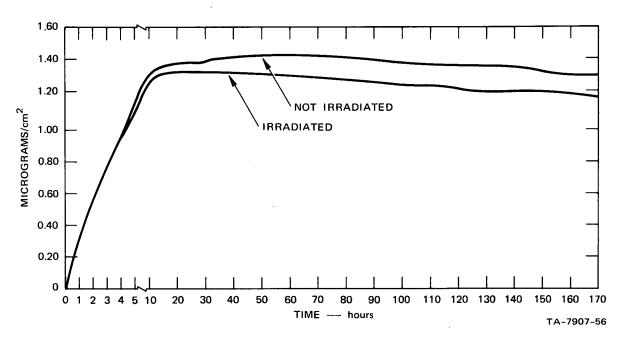


FIGURE 44 MICROBALANCE RESULTS—EMERSON AND CUMING, INC. STYCAST 1090

be heaviest on the irradiated side. For some unknown reason, Figure 44 indicates the opposite.

The quadrupole mass spectra of sample outgases and gases emitted from the heated chill-plate are weak and show nothing significant besides water.

### Epon 828 - Epoxy Adhesive, Shell Chemical Co.

Epon 828 was prepared using 100 parts Epon 828 resin with 50 parts Versamid 125. A 1-g sample was exposed for 217 hours to an irradiated thermal-vacuum test.

Reflectivity curves shown in Figure 45 and microbalance results presented in Figure 46 all show that the irradiated side of the mirror has the heaviest deposit. The irregularities in the microbalance curves were caused by poor temperature control of the chill-plate. The deposit is light and ranges from 0.01 to 0.03 micron in thickness.

Quadrupole mass spectra are very weak with water being the major component.

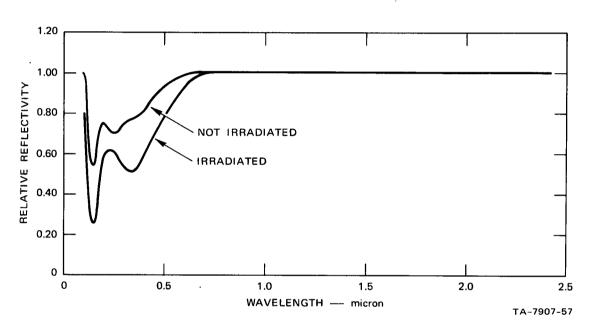


FIGURE 45 REFLECTIVITY OF A MIRROR CONTAMINATED WITH OUTGASES FROM SHELL EPON 828

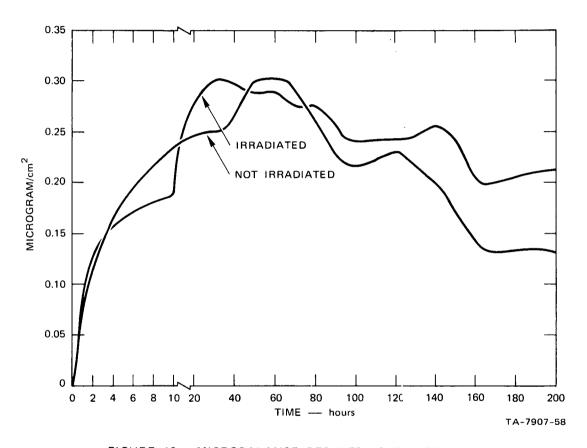


FIGURE 46 MICROBALANCE RESULTS—SHELL EPON 828

### FUTURE WORK

Before additional experiments are carried out, changes will be made to the existing equipment as called for in Modifications 1 and 2 of the contract. These include adapting a Cary Model 14 M spectrophotometer for making in situ measurements of the mirror's reflectivity from 0.110 to 2.50  $\mu m$ . These measurements will be made at 5, 20, and 100 hours with the mass accretions noted at those times. Also, the quadrupole mass spectrometer will be placed in line-of-sight with the outgassing sample.

Some of the materials that will be studied during the remainder of this study are given in Table II.  $\,$ 

Table II

# MATERIALS FOR FUTURE STUDY

Product	Manufacturer	Description
Eastman 910	Eastman Kodak	Adhesive
Armstrong X-81	Armstrong Products Co.	Epoxy adhesive
Epibond 153/9814	Furane Plastics, Inc.	Epoxy adhesive
3M Velvet Black 401-c-10	3M Company	Epoxy coating
G-683	General Electric Co.	Silicone grease
Scotchcast 502	3M Company	Epoxy potting compound
"O" ring	Porter Seal	Teflon gasket
MS 28775-006	Parker Seal	Rubber O-ring
Silastic 55U	Dow Corning Co.	Silicone elastomer
Silastic 955	Dow Corning Co.	Silicone elastomer
Myštic 7100	The Borden Co.	Double-faced tape
Alclad 7075-T6	Alcoa	Aluminum flat sheet
Rexolite 2200	American Enka Corp.	Copper clad polystyrene
DC-6-127/1528	Flexo Corp.	Silicone coated Fiberglas
Humiseal 1B12	Columbia Technical Corp.	Acrylic coating